IS 9407 (1980): Light Magnesium Oxide for Rubber Industry
[PCD 13: Rubber and Rubber Products]

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(Reaffirmed - 2012)

Indian Standard

SPECIFICATION FOR LIGHT MAGNESIUM OXIDE FOR RUBBER INDUSTRY

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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TO
IS 9407 : 1980 SPECIFICATION FOR LIGHT MAGNESIUM OXIDE FOR RUBBER INDUSTRY

[*Page* 4, *Table* 1, *Sl No.* (i), *col* 5] — Delete '3'.

(*Page* 8, *clause* C-1.1) — '*See* IS 265 : 1993*' *for* 'IS 265 : 1976*'.

(*Page* 8, *foot-note marked* *) — Substitute 'Hydrochloric acid — Specification (*fourth revision*)' for the existing.

(PCD 13)

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(*Continued on page* 14)

Indian Standard

SPECIFICATION FOR LIGHT MAGNESIUM OXIDE FOR RUBBER INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 5 January 1980, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 For manufacturing products of good quality, it is essential that all the raw materials used in its production are of proper quality. This standard prescribes the quality of light magnesium oxide which is used in the rubber industry as an activator.

0.3 This standard contains clauses **2.3** and **3.1** which call for agreement between the purchaser and the supplier.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for light magnesium oxide for rubber industry.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white, odourless, non-toxic free flowing powder.

2.2 The material shall also comply with the requirements given in Table 1.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS OF LIGHT MAGNESIUM OXIDE FOR RUBBER INDUSTRY

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix	Cl No. in IS : 7086 (Part I)-1973*
(1)	(2)	(3)	(4)	(5)
i)	Bulk density, g/ml	0·10 to 0·28	A	3
ii)	Sieve residue, percent by mass, *Max*		—	3
	a) Through 53-micron IS Sieve	0·2		
	b) Through 150-micron IS Sieve	0·01		
iii)	Relative density 27/27°C	3·4 ± 0·2	—	4
iv)	Moisture content, percent by mass, *Max*	1·0	—	7
v)	Matter soluble in water, percent by mass, *Max*	1·0	—	8
vi)	Matter insoluble in hydrochloric acid, percent by mass, *Max*	0·5	—	9
vii)	Loss on ignition, percent by mass, *Max*	5·0	—	10
viii)	Manganese (as Mn), percent by mass, *Max*	0·003	—	11
ix)	Copper (as Cu), percent by mass, *Max*	0·001	—	12
x)	Magnesium oxide, percent by mass, *Min*	94	B	—
xi)	Calcium oxide, percent by mass, *Max*	2	C	—
xii)	Iodine adsorption, mg of iodine/g of magnesium oxide, *Min*	100	D	—
xiii)	Aluminium and iron oxide, percent by mass, *Max*	1·0	E	—

*Methods of sampling and test for rubber compounding ingredients, Part I.

2.3 Compounding Test — If desired by the purchaser, the material may be compounded in polychloroprene rubber test recipe and the properties compared with the approved sample. The value for Mooney scorch obtained with the test sample shall not vary by more than 2 minutes from those obtained with the approved sample. The test recipe is given in Appendix F.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as agreed to between the purchaser and the supplier. Precautions shall be taken so as to protect it from moisture and moist air.

3.2 Marking — The following shall be marked on the package:

a) Name of the material;
b) Manufacturer's name and trade-mark, if any;
c) Net mass of contents; and
d) Month and year of manufacture.

3.3 BIS Certification Marking

The product may also be marked with Standard Mark.

3.3.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Sampling — The sampling shall be done as prescribed in 15 of IS : 7086 (Part I)-1973*.

4.2 Number of Tests and Criteria for Conformity — Tests for all characteristics shall be conducted on the composite sample. The lot shall be considered as conforming to the specification if the composite sample satisfies each of these requirements.

APPENDIX A

[*Table* 1, *Item* (i)]

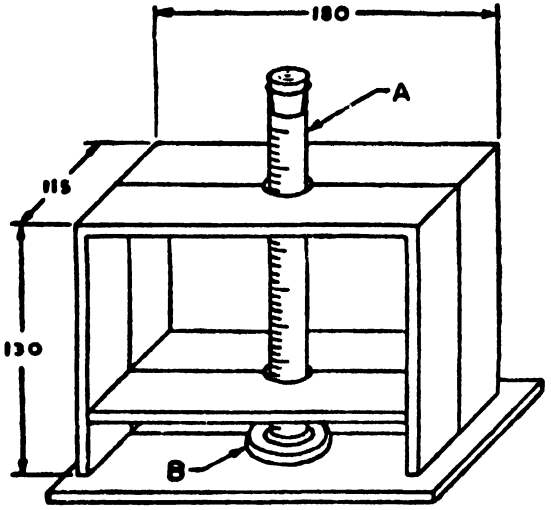
DETERMINATION OF BULK DENSITY

A-1. APPARATUS

A-1.1 Assemble the apparatus as shown in Fig. 1. The base of the measurement cylinder *A* shall be ground flat and the empty measuring cylinder *A* together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error, if any, of less than 1 ml.

*Methods for sampling and test for rubber compounding ingredients, Part I.

The distance between zero and 250-ml graduation on the measuring cylinder *A* shall be not less than 220 mm and not more than 240 mm. The distance between the flat-ground part of the base of measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height shall be 25 ± 2 mm.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

A-1.2 Rubber Base Pad — The rubber base pad *B* shall have a hardness of 42 to 50 IRHD.

A-1.3 Balance — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0·1 g.

A-2. PROCEDURE

A-2.1 Sieve about 40 g of the material through 250-micron IS Sieve on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder, and within one second lift it about 25 mm (taking care not to jerk the cylinder by knocking it against the upper stop) and let it drop. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During this operation give a gentle turn of about 10° in the clockwise direction to the cylinder

after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

A-3. CALCULATION

A-3.1 Calculate bulk density as follows:

$$\text{Bulk density, g/ml} = \frac{m}{V}$$

where

m = mass in g of the material taken for the test, and

V = volume in ml of the material after 50 taps.

APPENDIX B

[*Table* 1, *Item* (x)]

DETERMINATION OF MAGNESIUM OXIDE

B-1. REAGENTS

B-1.1 Standard Sulphuric Acid — 1 N.

B-1.2 Standard Sodium Hydroxide Solution — 1 N, freshly standardized.

B-1.3 Methyl Orange Indicator Solution — Dissolve 0·01 g of methyl orange in 100 ml of water.

B-2. PROCEDURE

B-2.1 Weigh accurately about 1 g of the freshly ignited material and transfer it to a 250-ml conical flask. Add into the flask about 20 ml of water and transfer with a pipette 25 ml of standard sulphuric acid. Cover the flask with a watch-glass and stir the flask carefully. Wash down the watch-glass and the sides of the conical flask with water and titrate the solution in the flask with standard sodium hydroxide solution using methyl orange indicator. Carry out a blank determination using the same quantities of the reagents.

B-3. CALCULATION

$$\text{Magnesium oxide, percent by mass of the ignited residue} = \frac{2{\cdot}016\,(V_1 - V_2)\,N}{M} - 0{\cdot}719A$$

where

V_1 = volume in ml of standard sodium hydroxide solution required for titration in the blank determination,

V_2 = volume in ml of standard sodium hydroxide solution required for titration in the test with the material,

N = normality of standard sodium hydroxide solution,

M = mass in g of the ignited material taken for the test, and

A = percentage by mass of calcium oxide as obtained in **C-3**.

APPENDIX C

[*Table* 1, *Item* (xi)]

DETERMINATION OF CALCIUM OXIDE

C-1. REAGENTS

C-1.1 Concentrated Hydrochloric Acid — *See* IS : 265-1976*.

C-1.2 Rectified Spirit — 95 percent (*v/v*).

C-1.3 Dilute Sulphuric Acid — 25 percent (*m/v*).

C-2. PROCEDURE

C-2.1 Dissolve about 0·5 g of freshly ignited material, accurately weighed, in concentrated hydrochloric acid added in small portions. Filter, if necessary. Add to the filtrate 100 ml of rectified spirit and 40 ml of dilute sulphuric acid and let stand overnight. If crystals of magnesium sulphate separate out, warm the mixture to about 50°C to dissolve them. Filter, wash with a mixture of two volumes of rectified spirit and one volume of sulphuric acid and ignite at 650°C. Weigh the residue.

*Specification for hydrochloric acid (*second revision*).

C-3. CALCULATION

$$\text{Calcium oxide, percent by mass of the ignited residue} = \frac{41{\cdot}18\,A}{M}$$

where

A = mass in g of the residue, and

M = mass in g of the ignited material taken for the test.

APPENDIX D

[*Table* 1, *Item* (xii)]

DETERMINATION OF IODINE ADSORPTION VALUE

D-0. GENERAL

D-0.1 The method of determining the iodine adsorption value is based on the adsorption of iodine by magnesium oxide sample in a non-aqueous solution. From the concentration difference of a standard iodine solution before and after exposure to the sample, the iodine adsoprtion value is calculated.

D-1. REAGENTS

D-1.1 Carbon Tetrachloride — free from sulphur or carbon disulphide.

D-1.2 Chloroform

D-1.3 Ethyl Alcohol — 95 percent (*v/v*).

D-1.4 Iodine — crystals.

D-1.5 Potassium Iodide Solution — Dissolve 5 g of potassium iodide in 1 litre of a solvent mixture containing 75 percent of 95 percent ethyl alcohol and 25 percent of distilled water by volume.

D-1.6 Sodium Hydroxide Solution — 1 N.

D-1.7 Dilute Sulphuric Acid — Dilute concentrated acid by adding 1 part of acid to 9 parts of water.

D-1.8 Starch Indicator Solution — Weigh 2·5 g of soluble starch on a small watch-glass and make this into a thin paste by adding a few millilitres of cold water. Pour this paste slowly into a beaker containing 500 ml of boiling distilled water. Stir the mixture gently while adding the paste to the water and continue stirring until a clear solution results. Boil for an additional 2 minutes after which the contents can be cooled on a

water-bath and transfer to a glass-stoppered bottle. A few drops of chloroform in the cooled starch solution will serve as a preservative.

D-1.9 Standard Potassium Iodate Solution — 0·05 N. Weigh approximately 1·784 g of potassium iodate to the nearest 0·1 mg and dissolve in distilled water and dilute to exactly 1 litre.

$$\text{Normality of potassium iodate} = \frac{M \times 0{\cdot}05}{1{\cdot}783\,5}$$

where M is actual mass of potassium iodate taken in g.

D-1.10 Standard Sodium Thiosulphate Solution — 0·05 N. Weigh 12·415 g of sodium thiosulphate pentahydrate and dissolve in about 500 ml of distilled water. Add 6 drops of 1 N sodium hydroxide, 2 or 3 drops of chloroform and dilute to 1 litre. This solution is standardized by titration against potassium iodate solution. Pipette 25 ml of standard 0 05 N potassium iodate solution into a 250-ml conical flask containing 50 ml of 0·03 N potassium iodide solution in ethyl alcohol and 5 ml of dilute sulphuric acid. Titrate immediately with sodium thiosulphate solution to be standardized until the solution in the flask exhibits a light yellow colour. At this point add 2 to 3 ml of starch indicator solution and continue the titration until the blue colour just disappears. Record the volume in millilitres of sodium thiosulphate solution required to reach the end point. Calculate the normality of sodium thiosulphate solution as follows:

$$\text{Normality of thiosulphate solution} = \frac{V_1 N}{V_2}$$

where

V_1 = volume in ml of standard potassium iodate solution taken,

N = normality of the potassium iodate solution, and

V_2 = volume in ml of the sodium thiosulphate solution consumed.

D-1.11 Standard Solution of Iodine in Carbon Tetrachloride — Add 12·7 g of iodine to a flask containing approximately 500 ml of carbon tetrachloride. Place the flask in a mechanical shaker and allow the flask to be agitated until complete solution is effected. This will require several hours as iodine crystals dissolve very slowly in carbon tetrachloride. When solution is complete dilute with more carbon tetrachloride to volume of 1 litre.

D-1.11.1 Pipette 20 ml of iodine solution in conical flask containing 50 ml of 0·03 N potassium iodide in alcohol. Titrate with standard 0·05 N sodium thiosulphate solution until the colour of the solution changes from light yellow to colourless. No starch indicator is necessary. Record the volume in millilitres of sodium thiosulphate solution required to reach the end point. The strength of the iodine solution is given by the formula:

$$\frac{V_1 N}{V_2}$$

where

V_1 = volume in ml of the sodium thiosulphate required to reach the end point,

N = normality of the sodium thiosulphate solution, and

V_2 = volume in ml of the iodine solution taken.

D-2. PROCEDURE

D-2.1 Weigh 2·000 ± 0·005 g of the sample. Transfer the sample to a clean, dry 200 ml glass-stoppered bottle. Add 100 ± 0·2 ml of standardized 0·1 N iodine in carbon tetrachloride. Stopper the bottle and clamp it in a suitable shaking device. Allow it to shake vigorously for exactly 30 minutes. Allow the suspension to settle for 5 minutes. At this time pipette a 20-ml aliquot of the clear solution into 250-ml conical flask containing 50 ml of 0·03 N potassium iodide in ethyl alcohol. Do not pipette any solids. Titrate the aliquot with standard 0·05 N sodium thiosulphate. A sharp end point can be obtained without the use of starch indicator.

D-3. CALCULATION

$$\text{Iodine adsorption value} = 2{\cdot}5 \times 127 \, (V_1 - V_2) \, N$$

where

V_1 = volume in ml of standard sodium thiosulphate solution equivalent to 20 ml of original iodine solution,

V_2 = volume in ml of standard sodium thiosulphate solution equivalent to 20 ml of iodine solution after exposure to magnesium oxide sample, and

N = normality of sodium thiosulphate solution.

APPENDIX E

[*Table* 1, *Item* (xiii)]

DETERMINATION OF ALUMINIUM OXIDE AND IRON OXIDE

E-1. REAGENTS

E-1.1 Ammonium Chloride

E-1.2 Ammonium Hydroxide Solution — 50 percent (*v/v*).

E-1.3 Ammonium Nitrate Solution — 2 percent (*m/v*).

E-1.4 Dilute Hydrochloric Acid — 50 percent (*v/v*).

E-1.5 Concentrated Nitric Acid

E-2. PROCEDURE

E-2.1 Weigh accurately about 2 g of the light magnesium oxide sample in a 400-ml beaker, add 50 ml of 50 percent hydrochloric acid, boil, dilute to 100 ml and filter. Wash the residue 5 times with 10 ml portions of hot distilled water. (The residue may be ignited and weighed for the estimation of insoluble siliceous matter.) To the filtrate add 2 g of ammonium chloride, a few drops of concentrated nitric acid, boil for 10 minutes and cool. Add ammonium hydroxide until just alkaline, bring to boil, allow to stand for 10 minutes and filter the precipitate. Dissolve the residue into a new beaker by washing with 50 ml of hydrochloric acid followed by 5 hot distilled water washes of 10 ml each. More of hydrochloric acid and water may be used if the residue is undissolved. Add 2 g of ammonium chloride and re-precipitate with ammonium hydroxide, boil and filter. Wash with 5 portions of ammonium nitrate 10 ml each. Transfer residue to a pre-ignited tared crucible, ignite, cool in a desiccator and weigh.

E-3. CALCULATION

$$\text{Aluminium oxide and iron oxide, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of the residue, and

M = mass in g of the sample.

APPENDIX F

(*Clause* 2.3)

RECIPE FOR COMPOUNDING TEST

F-1. Test Recipe — The following test recipe is recommended for checking performance test requirements:

Ingredient	*Parts by Mass*
Polychloroprene rubber	100
Stearic acid (*see* IS : 1675-1971*)	1
Semi-reinforcing furnace (SRF) carbon black	30
Zinc oxide (*see* IS : 3399-1973†)	5
Magnesium oxide	4

*Specification for stearic acid, technical (*first revision*).
†Specification for zinc oxide for rubber industry (*first revision*).

(*Continued from page* 2)

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